

**ADDENDUM TO**  
**Certification of a Resin-Bonded Glass Fibre Board for**  
**Thermal Conductivity between – 10 °C and + 50 °C**  
**IRMM-440**

**Report**  
**EUR 19572 EN**



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#### Abstract

This Addendum to report EUR 19572 EN describes the re-assessment of the stability of IRMM-440 and the characterisation of the thermal conductivity at lower temperature in the light of new data.

The available data show that, as long as the boards are not physically damaged, IRMM-440 is stable for at least 10 years and can be used at least 100 times.

Data from an international intercomparison were used for the characterisation of thermal conductivity at low temperatures. All participating laboratories had demonstrated their competence and the results at -10, 0 and 10 °C agree with the certified values.

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## Summary

This Addendum to report EUR 19572 EN describes the re-assessment of the stability of IRMM-440 and the characterisation of the thermal conductivity at lower temperature in the light of new data.

The available data show that, as long as the boards are not physically damaged, IRMM-440 is stable for at least 10 years and can be used at least 100 times.

Data from an international intercomparison were used for the characterisation of thermal conductivity at low temperatures. All participating laboratories had demonstrated their competence and the results at -10, 0 and 10 °C agree with the certified values. Based on these data, the following indicative values were assigned:

	Thermal conductivity	
	Indicative value <sup>2)</sup> [W/(m·K)]	Uncertainty <sup>3)</sup> [%]
Thermal conductivity <sup>1)</sup> as a function of test temperature T in °C for -170 °C ≤ T < -10 °C	$22.95 \cdot 10^{-2} + \frac{T}{^{\circ}\text{C}} \cdot 1.08 \cdot 10^{-4} + \frac{T^2}{(^{\circ}\text{C})^2} \cdot 2 \cdot 10^{-8}$	5
<p>1) Thermal conductivity as determined by the guarded hot plate technique (ISO 8302).</p> <p>2) The indicative value is obtained by fitting of an unweighted second-order polynomial through data obtained by 5 laboratories. The value is traceable to the International System of Units (SI).</p> <p>3) Relative expanded uncertainty (k=2.78) at about 95 % confidence level over the temperature range [-10, -170] °C. The uncertainty is deduced from the uncertainties of thermal conductivity measurements at the 5 participating laboratories, and from the uncertainty due to the fit of thermal conductivity versus temperature.</p>		



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# Glossary

$a_0, a_1, a_2$	Parameters of the second order regression
ANOVA	Analysis of variance
BCR <sup>®</sup>	One of the trademarks of CRMs owned by the European Commission; formerly Community Bureau of Reference
CRM	Certified reference material
EC	European Commission
EU	European Union
GUM	Guide to the Expression of Uncertainty in Measurements
IRMM	Institute for Reference Materials and Measurements of the JRC
ISO	International Organization for Standardization
JRC	Joint Research Centre of the European Commission
$k$	Coverage factor
$n$	Number of replicates per unit
$N$	Number of samples (units) analysed
n.a.	Not applicable
RM	Reference material
$s$	Standard deviation
$s_{bb}$	Between-unit standard deviation; an additional index "rel" is added when appropriate
$T$	Temperature
$u$	standard uncertainty
$U$	expanded uncertainty
$u_c$	combined standard uncertainty; an additional index "rel" is added as appropriate
$u_{char}$	Standard uncertainty of the material characterisation; an additional index "rel" is added as appropriate
$u_{CRM}$	Combined standard uncertainty of the certified value; an additional index "rel" is added as appropriate
$U_{CRM}$	Expanded uncertainty of the certified value; an additional index "rel" is added as appropriate
$\bar{x}$	Arithmetic mean
$\nu$	Degrees of freedom



# 1 Introduction

## 1.1 Background

IRMM-440 was certified in 2000 [1] and based on tests conducted by IRMM, a shelf life of 3 years or 30 repeated measurements was assigned. There was a desire by users of this material to prolong the shelf life and/or allow for higher number of tests per sample. In addition, increasing environmental awareness increased the interest of measurement of thermal conductivity at lower temperature.

Several users of IRMM-440, led by the Expert Group of the Keymark Scheme for thermal conductivity ([www.key-mark.org](http://www.key-mark.org)), provided additional data to allow re-assessment of stability and the thermal properties at low temperatures.

## 2 Participants

### 2.1 Data review and value assignment

European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE

(accredited to ISO Guide 34 for production of certified reference materials, BELAC No. 268-RM)

### 2.2 Additional data for the assessment of stability

Forschungsinstitut für Wärmeschutz e.V.(FIW), München, DE

National Physical Laboratory (NPL), Teddington, UK

Rockwool International, Hedehusene, DK

### 2.3 Characterisation of thermal conductivity at low temperatures

Laboratoire National d'Essais (LNE), Division Energie et Produits pour la Construction, Trappes Cedex, FR

National Physical Laboratory (NPL), Teddington, UK

Forschungsinstitut für Wärmeschutz (FIW), Gräfelfing, DE

Dipartimento di Fisica Tecnica (DFT), Padova, IT

Instytut Mechanizacji Budownictwa i Górnictwa Skalnego (IMBiGS), Warszawa, PL

## 3 Homogeneity

A key requirement for any reference material is the equivalence between the various units. In this respect, it is relevant whether the variation between units is significant compared to the uncertainty of the certified value. In contrast to that it is not relevant if this variation between units is significant compared to the analytical variation. Consequently, ISO Guide 34 requires RM producers to quantify the between unit variation. This aspect is covered in between-unit homogeneity studies.

Homogeneity of thickness, density and thermal conductivity at 20 °C had been ascertained in the course of the original certification report [1]. Each participant in the characterisation of thermal conductivity at low temperatures used two different specimens. No difference was found between laboratories and specimens, demonstrating that the thermal conductivity is also homogeneous at temperatures down to -170 °C.

## 4 Stability

Stability testing is necessary to establish conditions for storage (long-term stability) as well as conditions for dispatch to the customers (short-term stability). During transport, especially in summer time, temperatures up to 60 °C could be reached and stability under these conditions must be demonstrated if transport at ambient temperature will be applied.

IRMM organised a series of tests demonstrating that 30 heating cycles do not affect the stability of the material. To allow a long-term assessment of the stability of the material, the following data were evaluated:

### 4.1 Data on BCR-64

BCR-64 was the predecessor of IRMM-440 and was certified in 1982 [2]. As BCR-64 consists of the same type of resin-bonded glass-fibre board, data on this material provide useful information on the stability of IRMM-440.

One sample of BCR-64 was tested by FIW 10 times between 1983 and mid-2005. The results never deviated by more than 1 % from the certified value (see Figure 1), which is within the certified range (1.2-2.4 %, depending on the test temperature). These data demonstrate that the thermal conductivity of this type of material is, given adequate storage, stable for more than 20 years.

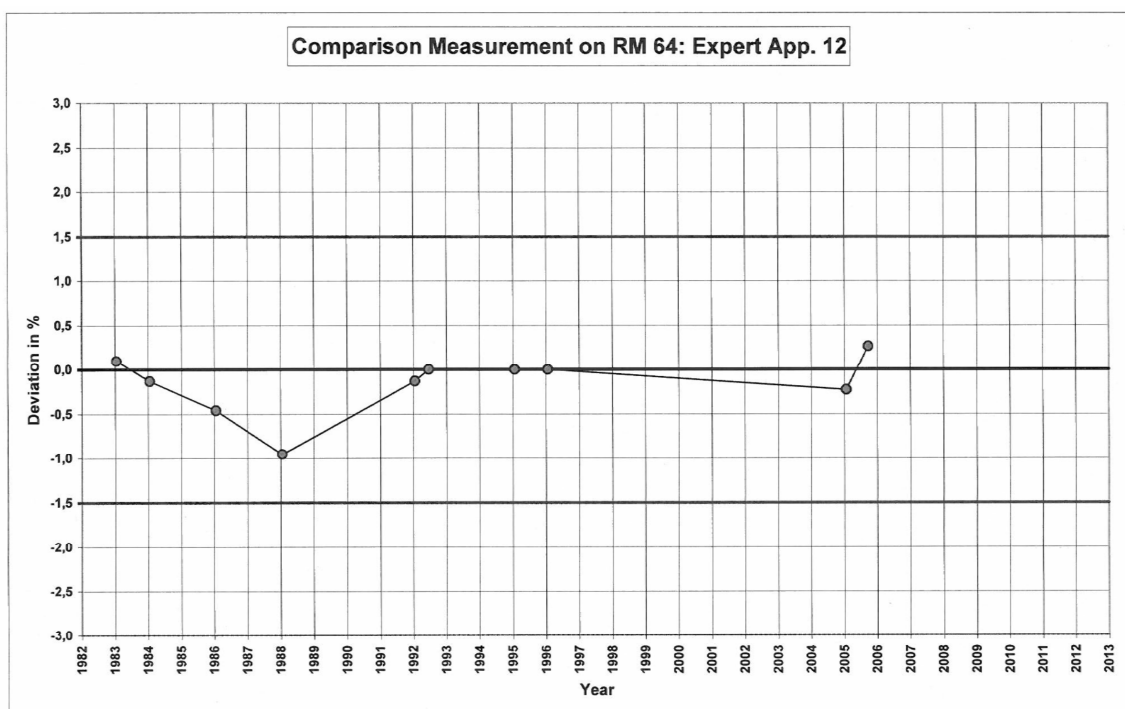


Figure 1: Stability data for BCR-64 provided by FIW

## 4.2 Data on IRMM-440

### 4.2.1 Results obtained by NPL

NPL repeatedly tested one sample of IRMM-440, using three different guarded hot plate (GHP) apparatuses at temperatures between -5 and 50 °C. 98 measurements were performed in total over a period of 12 years. Deviations of the certified value ranged from 0.4 to -1.1 % (see Figure 2). 4 of the 98 results were outside the certified range of 0.00028 W/(m.K), which is within the expected range of a 95 % confidence interval.

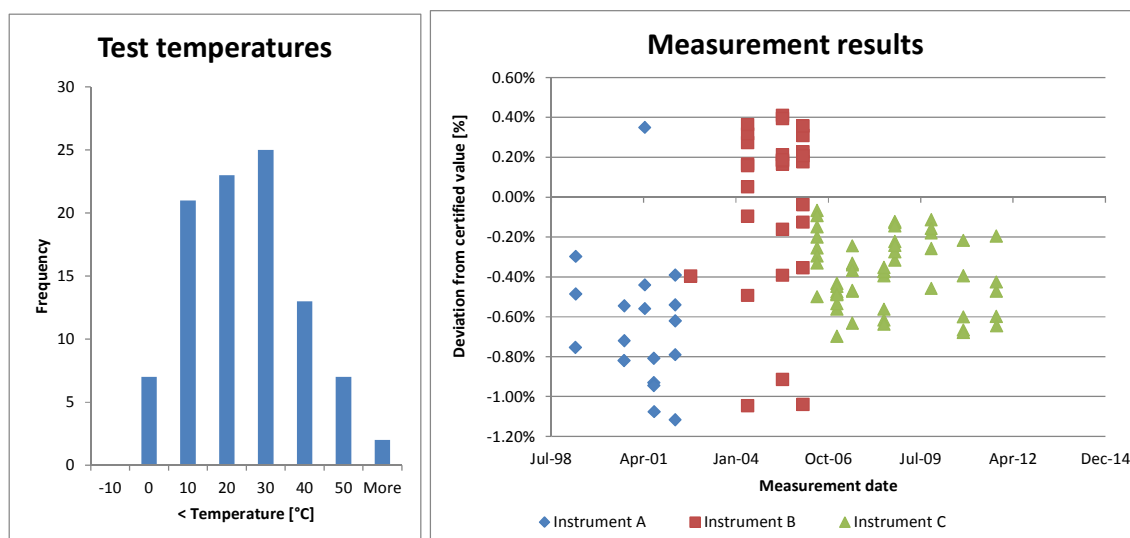


Figure 2: Stability data on IRMM-440 sample B31b provided by NPL. The distribution of the test temperatures is given on the left, whereas the deviation from the certified values is given on the right.

The measurement temperatures were spread well over the complete temperature range. The results themselves show clear dependency on the instrument used, with especially results for instrument B shifted towards higher values.

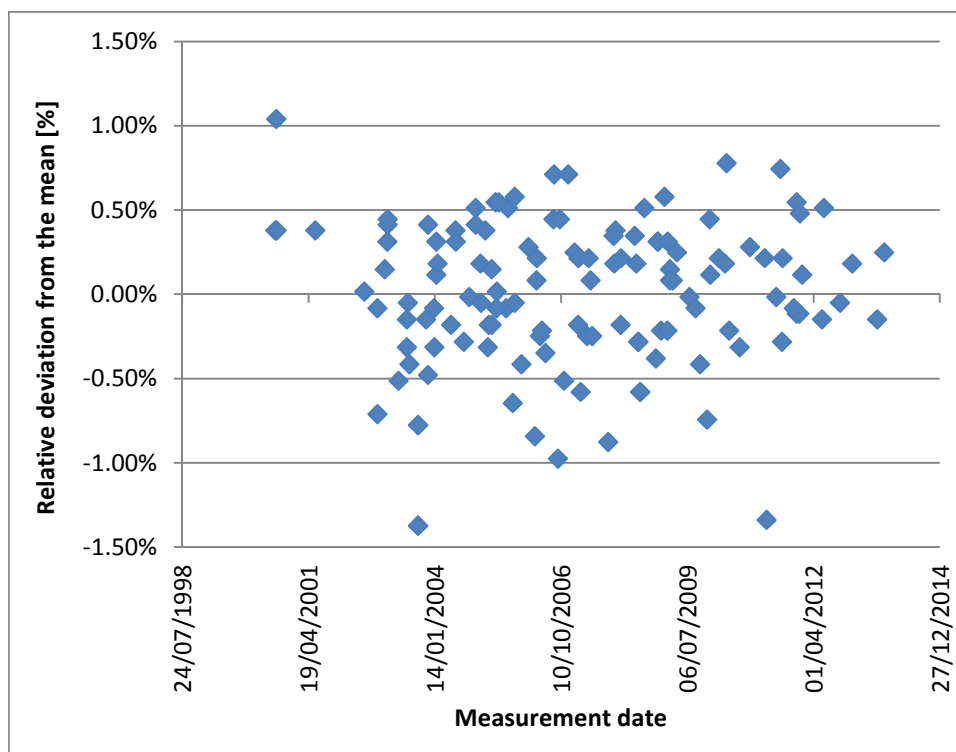
Data were corrected for this shift according to Equation 1

$$x_{corr} = x_i - \bar{x} \quad \text{Equation 1}$$

With  $x_{corr}$  the corrected value,  $x_i$  the measurement result and  $\bar{x}$  the average result for a given instrument. The corrected results were subjected to a trend test and no significant trend was detected on a 95 % confidence level.

#### 4.2.2 Data provided by Rockwool International

Rockwool International tested one specimen on in total 127 occasions at different sites using in total 28 different instruments over a period of 13 years (August 2000 – October 2013). Test temperature was in all cases 10 °C. The data are shown in Figure 3. A regression analysis of the data showed no significant trend on a 95 % confidence level.



*Figure 3: Stability data on IRMM-440 provided by Rockwool International. The data show the relative deviation of the measurement result of the mean over all results.*

#### 4.3 Conclusion

The data available demonstrate that, provided samples are not physically damaged and stored properly, IRMM-440 is stable over extended periods and frequent measurement cycles. The new shelf life for IRMM-440 is therefore set as the longer of 10 years after sales or 100 measurements.

Based on the care of handling and storage conditions, samples may be stable for even longer, but this is beyond the control of IRMM. Customers can demonstrate the stability of their individual sample beyond the stated shelf lives by performing regression analyses in the same way as described in 4.2.2.

## 5 Characterisation

The material characterisation was based on an intercomparison of expert laboratories, i.e. the thermal conductivity of the material was determined in different laboratories using the guarded hot plate technique. This approach aims at randomisation of laboratory bias, which reduces the combined uncertainty.

In order to assess the thermal conductivity at low temperatures, LNE organised a measurement campaign involving three laboratories and combined the results with data from DFT presented in the original certification report as well as data from NPL that were published independently [3]. The evaluation was presented at the 32<sup>nd</sup> international thermal conductivity conference [4].

### 5.1 Selection of participants

LNE, FIW and NPL laboratories have demonstrated their competence in the determination of thermal conductivity in the Keymark system.

### 5.2 Study setup

Each laboratory received used 2 of its units of IRMM-440 and was requested to provide thermal conductivity measurements from at least -150 °C to +10 °C. The overlap between the new and the certified values, which range from -10 °C to +50 °C, allow demonstration of proper function of each measurement instrument.

### 5.3 Methods used

All laboratories used the a Guarded Hot Plate apparatus in a double-sided mode (heat flow through a pair of specimens). Guarded Hot Plates designed to make measurements at low temperature have cold plates that are cooled with a cooling fluid (in general liquid nitrogen) and heated to a specified temperature with an electrical heater (Figure 1). The heater plate is designed like a standard Guarded Hot Plate, but with a particular attention to the deformation of plate due to temperature. The main instrument parameters are summarised in Table 1.

**Table 1:** Instrument parameters of the various instruments

Parameter	L1	L2	L3	L4	L5
Plate, mm	340x340	500x500	305x305	300x300	300x300
Meter plate, mm	200x200	247x247	152.5 x 152.5	148x158	148x148
Guard-center gap, mm	2	3	2	2	2
Plate emittance	>0.9	>0.9	>0.89	not given	>0.9
Temperature sensor	T type	PT 100	T type	not given	PT 100
Operation mode	2-sided	2-sided	2-sided	2-sided	2-sided

## 5.4 Evaluation of results

All individual results of the participants, are displayed in Figure 4.

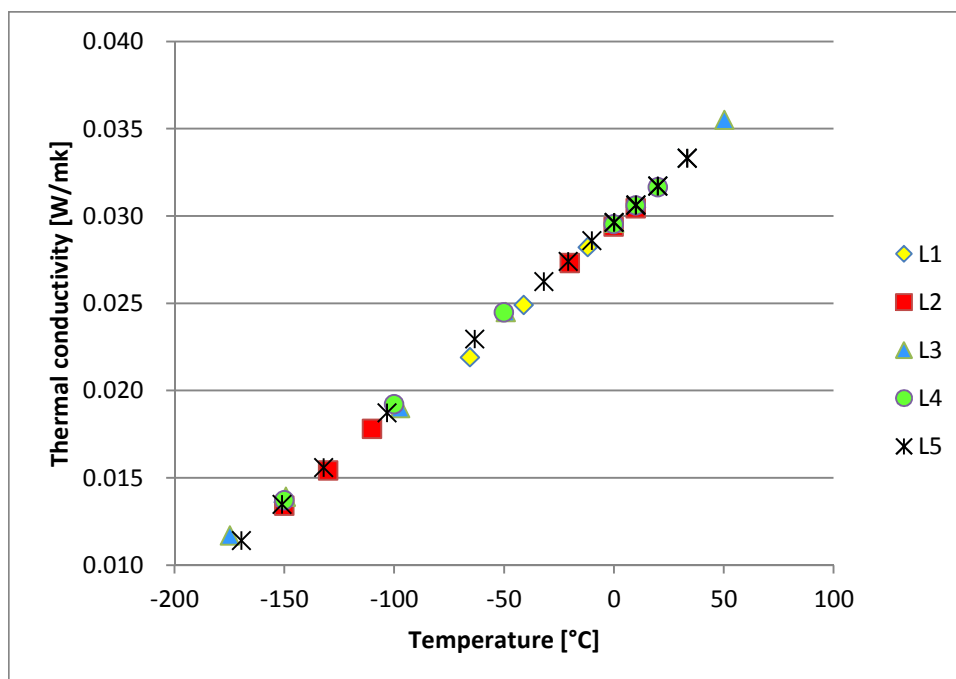


Figure 4: Data used for the assessment of the thermal conductivity of IRMM-440 at low temperatures

A comparison of thermal conductivity data must be performed at defined temperatures. Data of each laboratory were fitted with a 2<sup>nd</sup> order polynomial curve and thermal conductivities at -150, -100, -50, -10, 0, +10 °C were interpolated. Also data for -170 °C were calculated for those two laboratories that measured down to this temperature.

### 5.4.3 Technical evaluation

Regression lines were calculated for the individual data for each laboratory and the interpolated data were checked for agreement with the certified values at -10, 0 and 10 °C. All values were in agreement with the certified. In fact, apart from the interpolated value for +10 °C of L3, all values were even within the certified interval. This agreement demonstrates the technical validity of all results and the proper calibration of the instruments used at least within the certified range. The agreement also confirms yet again the proficiency of the laboratories involved.

### 5.4.4 Statistical evaluation

The low number of datasets means that a numerical statistical evaluation is not possible. A visual evaluation was therefore performed.

The interpolated data at -170, -150, -100, -50, -10, 0 and 10 °C were compared graphically with the mean value. A plot showing relative deviations for each laboratory and temperature from the mean value was prepared (see Figure 5). It is clear that data below -10 °C scatter more than in the range -10 to 10 °C, but no laboratory shows consistently extreme values. Therefore, all data were accepted for evaluation on statistical grounds.

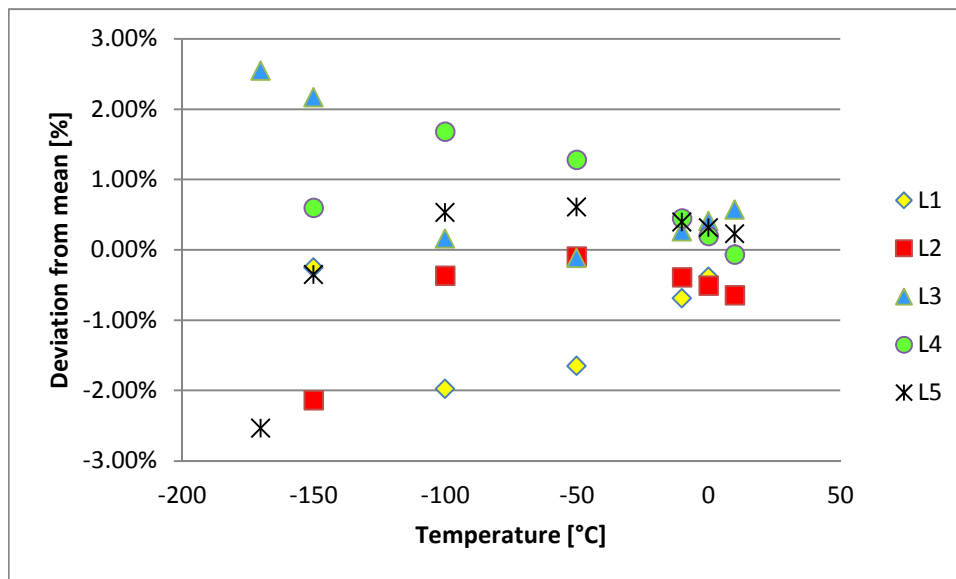


Figure 5: Relative deviation of interpolated data from the mean of all laboratories

## 5.5 Regression analysis

The average interpolated thermal conductivity data at -150, -100 and -50 (and, for L3 and L5 also of -170) were calculated from the data of all laboratories. The certified value was used for the thermal conductivity at -10 °C.

A second order polynomial  $\lambda = a_0 + a_1 \cdot T + a_2 \cdot T^2$  was fitted using the least squares method and the regression parameters shown in Table 2 were obtained.

**Table 2:** Regression parameters for the thermal conductivity of IRMM-440 at Temperatures below -10 °C

Parameter	Value	Uncertainty
$a_0$	$2.945571 \cdot 10^{-02}$	$9.0453 \cdot 10^{-05}$
$a_1$	$1.076644 \cdot 10^{-04}$	$2.5257 \cdot 10^{-06}$
$a_2$	$1.799309 \cdot 10^{-08}$	$1.3486 \cdot 10^{-08}$

The constant and linear terms are statistically significant. The second order term is not statistically significant, but was nevertheless kept as experience with these measurements indicating a non-linear relationship.

The result obtained for -10 °C agrees with the certified value, demonstrating the correctness of the composed result.

## 5.6 Estimation of $u_{\text{char}}$

For the uncertainty of characterisation, the uncertainty of the regression line itself as well as the scatter of the results received from the laboratory was taken into account. Although the uncertainty of the regression line already contains a component due to the variation of individual results, the regression is based on averages on points coming from regression lines per laboratory, which themselves average over several points. Using only the uncertainty of the regression line was therefore thought to underestimate the assigned uncertainty.

The uncertainty of the regression line was estimated as the standard deviation of the residuals of the 5 temperatures around the regression line.

The uncertainty due to the scatter of results was estimated as follows:

- For -150, -100 and -50 °C as the standard deviation of the results from the 5 regression lines of the individual laboratories.
- For -170 °C (where only two laboratories had submitted data), the range of the two results was assumed to be the limits of a rectangular distribution and the corresponding uncertainty was therefore obtained by dividing the range of the two results by two times  $\sqrt{3}$ .

The various uncertainty components and  $u_{\text{char}}$  are listed with their respective degrees of freedom in Table 3.

**Table 3:** Uncertainty budget of  $u_{\text{char}}$ . The degrees of freedom were calculated using the Welch-Satterthwaite equation [5].

Parameter Parameter	uncertainty of the regression line		uncertainty due to scatter of results		combined uncertainty of characterisation		
	$u$ [W/(m·K)]	$\nu$	$u$ [W/(m·K)]	$\nu$	$u_{\text{char}}$ [W/(m·K)]	$u_{\text{char,rel}}$ [%]	$\nu$
-50 °C	$5.3791 \cdot 10^{-5}$	4	$2.63706 \cdot 10^{-4}$	4	$2.69136 \cdot 10^{-4}$	1.12%	4
-100 °C	$5.3791 \cdot 10^{-5}$	4	$2.52726 \cdot 10^{-4}$	4	$2.58387 \cdot 10^{-4}$	1.37%	4
-150 °C	$5.3791 \cdot 10^{-5}$	4	$2.13842 \cdot 10^{-4}$	4	$2.20504 \cdot 10^{-4}$	1.61%	4
-170 °C	$5.3791 \cdot 10^{-5}$	4	$1.72223 \cdot 10^{-4}$	$\infty$	$1.80428 \cdot 10^{-4}$	1.55%	$\approx 500$

## 6 Value Assignment

As there were some deviations from the requirements of ISO Guide 34 (two datasets were already known to the laboratories providing the other three) as well as due to the low overall number of datasets (4), indicative rather than certified values were assigned for the thermal conductivity below -10 °C. According to IRMM's policy, indicative values are values where either the uncertainty is deemed too large or where too few independent datasets were available to allow certification. Uncertainties are evaluated according to the same rules as for certified values.

The regression line as obtained in 5.5 was assigned as indicative value for the range  $-170 \text{ °C} \leq T < -10 \text{ °C}$ :

$$2.95 \cdot 10^{-2} + \frac{T}{\text{°C}} \cdot 1.08 \cdot 10^{-4} + \frac{T^2}{(\text{°C})^2} \cdot 2 \cdot 10^{-8}$$

$\lambda$  thermal conductivity in W/(m·K)

$T$  temperature in °C

The assigned uncertainty consists in principle of uncertainties related to characterisation,  $u_{\text{char}}$ , potential between-unit inhomogeneity,  $u_{\text{bb}}$  and potential degradation during transport ( $u_{\text{sts}}$ ) and long-term storage,  $u_{\text{lis}}$  (Section 4). As the contributions of homogeneity and stability were found to be negligible (see sections 3, 4), the assigned uncertainty consists only of  $u_{\text{char}}$ .

The relative uncertainties listed in Table 3 are approximately constant. Therefore, a constant relative uncertainty of 5 %, based on the largest relative uncertainty in Table 3 and a coverage factor of 2.78, reflecting 4 degrees of freedom was assigned.



## 7 Metrological traceability and commutability

### 7.1 Metrological traceability

#### Identity

The thermal conductivity according to the guarded hot-plate method is a method-defined measurand and can only be obtained by following the procedure specified in ISO 8302. Adherence to this procedure was confirmed by agreement of the laboratories' results with the assigned value for the CRM at higher temperatures. The assigned value is therefore operationally defined by ISO 8302.

#### Quantity value

Traceability of the obtained results is based on the traceability of all relevant input factors. Instruments in individual laboratories were verified and their influence parameters calibrated with tools ensuring traceability to the SI. Consistency in the interlaboratory comparison and agreement with the certified values demonstrates that all relevant input factors were covered. As the assigned values are combinations of agreeing results individually traceable to the International System of Units (SI), the assigned quantity value themselves is traceable to the SI as well.

### 7.2 Commutability

Many measurement procedures include one or more steps, which are selecting specific (or specific groups) of analytes from the sample for the subsequent steps of the whole measurement process. Often the complete identity of these 'intermediate analytes' is not fully known or taken into account. Therefore, it is difficult to mimic all the analytically relevant properties of real samples within a CRM. The degree of equivalence in the analytical behaviour of real samples and a CRM with respect to various measurement procedures (methods) is summarised in a concept called 'commutability of a reference material'. There are various definitions expressing this concept. For instance, the CSLI Guideline C-53A [6] recommends the use of the following definition for the term *commutability*:

"The equivalence of the mathematical relationships among the results of different measurement procedures for an RM and for representative samples of the type intended to be measured."

The commutability of a CRM defines its fitness for use and, thus, is a crucial characteristic in case of the application of different measurement methods. When commutability of a CRM is not established in such cases, the results from routinely used methods cannot be legitimately compared with the certified value to determine whether a bias does not exist in calibration, nor can the CRM be used as a calibrant.

As IRMM-440 was produced in the same way as normal insulation plate, it is commutable to other materials of this kind.

## 8 Acknowledgments

The authors would like to thank Alain Koenen (LNE), Erik Rasmussen (Rockwool International), Clark Stacey (NPL), Roland Schreiner (FIW), Grażyna Swólek (IMBiGS) for providing the data and their fruitful discussions as well as Yannic Ramaye and Robert Koeber (IRMM) for the reviewing of the certification report.

## 9 References

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## 10 Annex

### 10.1 Annex 1: Data submitted by the laboratories

Laboratory	Temperature [°C]	$\lambda$ [W/(m·K)]	Regression parameters $\lambda=a_0+a_1\cdot T+a_2\cdot T^2$	
L1	10.2	0.0305	$a_0$	$2.9425\cdot 10^{-2}$
	-11.9	0.0282	$a_1$	$1.1704\cdot 10^{-4}$
	-41	0.0249	$a_2$	$7.6992\cdot 10^{-8}$
	-65.4	0.0219	$r^2$	0.999
	-150.3	0.0136		
L2	10	0.03044	$a_0$	$2.9389\cdot 10^{-2}$
	0	0.02937	$a_1$	$1.0390\cdot 10^{-4}$
	-20	0.0273	$a_2$	$-2.0400\cdot 10^{-8}$
	-110	0.0178	$r^2$	1.000
	-130	0.0154		
	-150	0.0134		
L3	50.4	0.0355	$a_0$	$2.9659\cdot 10^{-2}$
	0.2	0.0296	$a_1$	$1.1327\cdot 10^{-4}$
	-49.1	0.0245	$a_2$	$5.6152\cdot 10^{-8}$
	-97.2	0.019	$r^2$	1.000
	-149.2	0.0139		
	-174.7	0.0117		
L4	20	0.03165	$a_0$	$2.9597\cdot 10^{-2}$
	10	0.0306	$a_1$	$1.0075\cdot 10^{-4}$
	0	0.02953	$a_2$	$-3.4060\cdot 10^{-8}$
	-50	0.02447	$r^2$	1.000
	-100	0.01922		
	-150	0.0137		
L5	33.45; 33.41	0.03331; 0.03329	$a_0$	$2.9632\cdot 10^{-2}$
	20.09; 20.05	0.03171; 0.0317	$a_1$	$1.0610\cdot 10^{-4}$
	10.13; 10.06	0.03063; 0.03061	$a_2$	$-5.7009\cdot 10^{-9}$
	0.22; 0.08	0.02963; 0.02962	$r^2$	1.000
	-9.96	0.02858		
	-20.76	0.02739		
	-31.77	0.02624		
	-63.22	0.02295		
	-103.25	0.01871		
	-132.07	0.01557		
	-150.98	0.01347		
	-169.54	0.01141		

## 10.2 Annex 2: Interpolated values used for value assignment

T	L1	L2	L3	L4	L5
[°C]	$\lambda$ [W/(m·K)]	$\lambda$ [W/(m·K)]	$\lambda$ [W/(m·K)]	$\lambda$ [W/(m·K)]	$\lambda$ [W/(m·K)]
10	0.030603	0.030425	0.030798	0.030601	0.030692
0	0.029425	0.029389	0.029659	0.029597	0.029632
-10	0.028263	0.028347	0.028532	0.028586	0.02857
-50	0.023766	0.024142	0.024136	0.024474	0.024313
-100	0.018491	0.018794	0.018894	0.019181	0.018965
-150	0.013601	0.013344	0.013932	0.013718	0.013588
-170			0.012026		0.01143

European Commission

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